

Experimenting with At-Home General Chemistry Laboratories During the COVID-19 Pandemic

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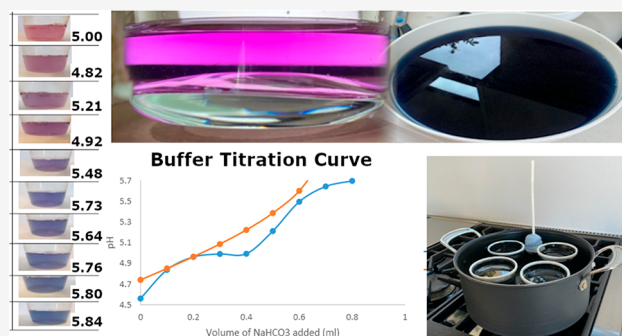
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ABSTRACT: During the COVID-19 pandemic, an at-home laboratory program was created and implemented for a section of the general chemistry course at the University of Southern California. The experiments were designed to only utilize safe household items and no special equipment. These laboratory activities, spanning over 4 weeks, focused on concepts usually covered in the final one-third of our second-semester chemistry laboratory, including pH, acid–base titrations, buffers, solubility, phase equilibria, and thermodynamics. In this article, we describe the design of the laboratories and our experience with this experiment, while also providing an assessment on how similar activities could be integrated profitably into a regular general chemistry course.

KEYWORDS: First-Year Undergraduate/General, Laboratory Instruction, Inquiry-Based/Discovery Learning, Hands-On Learning/Manipulatives, Acids/Bases, pH, Titration/Volumetric Analysis, Equilibrium, Precipitation/Solubility, Thermodynamics



During the COVID-19 pandemic in spring 2020, most universities in the United States were forced to switch to an online teaching modality. At the University of Southern California, this transition occurred after spring break, March 23rd, 2020. On the general chemistry (gen chem) course calendar, there were 6 weeks of lectures remaining in the spring semester and four laboratories to be completed.

CHEM-108 is a chemistry and biochemistry majors' second-semester gen chem class, with an enrollment of 44 students, in three lab sections, each with a teaching assistant (TA). Faced with this transition, the course faculty and the TAs contemplated various continuity options for the laboratories, ranging from canceling laboratories all together, to hosting virtual computer-based laboratories, to having the TAs perform the experiments on videos and the students analyzing data at home. At the end, the laboratory component of the class was deemed sufficiently essential to the learning objectives of the course that we wanted the students to earn a significant lab experience despite not being able to conduct the laboratories on-campus. Toward this purpose, we designed a series of at-home experiments to teach the final one-third of the laboratories, covering the following concepts: (I) pH, (II) acid–base titrations, (III) buffers, (IV) solubility, (V) phase equilibria, and (VI) thermodynamics.

DESIGNING THE EXPERIMENTS

The four principal elements of our at-home lab design were as follows: (1) presents no safety issues at home, (2) requires no special equipment, (3) delivers a genuine chemistry laboratory experience, and (4) directly interfaces with concepts I through VI covered in the lectures. The experiments consisted of these components:

- acid–base neutralization,
- titrations,
- pH measurements,
- designing buffers,
- assessing buffer capacities,
- measuring solubilities, and
- a van't Hoff analysis for measuring ΔH° and ΔS° .

While there have been many discussions about what the core contents of a gen chem course should be,^{1–5} the role of the laboratory has also been debated.^{6–10} At the authors' institution, the laboratories have traditionally run parallel to,

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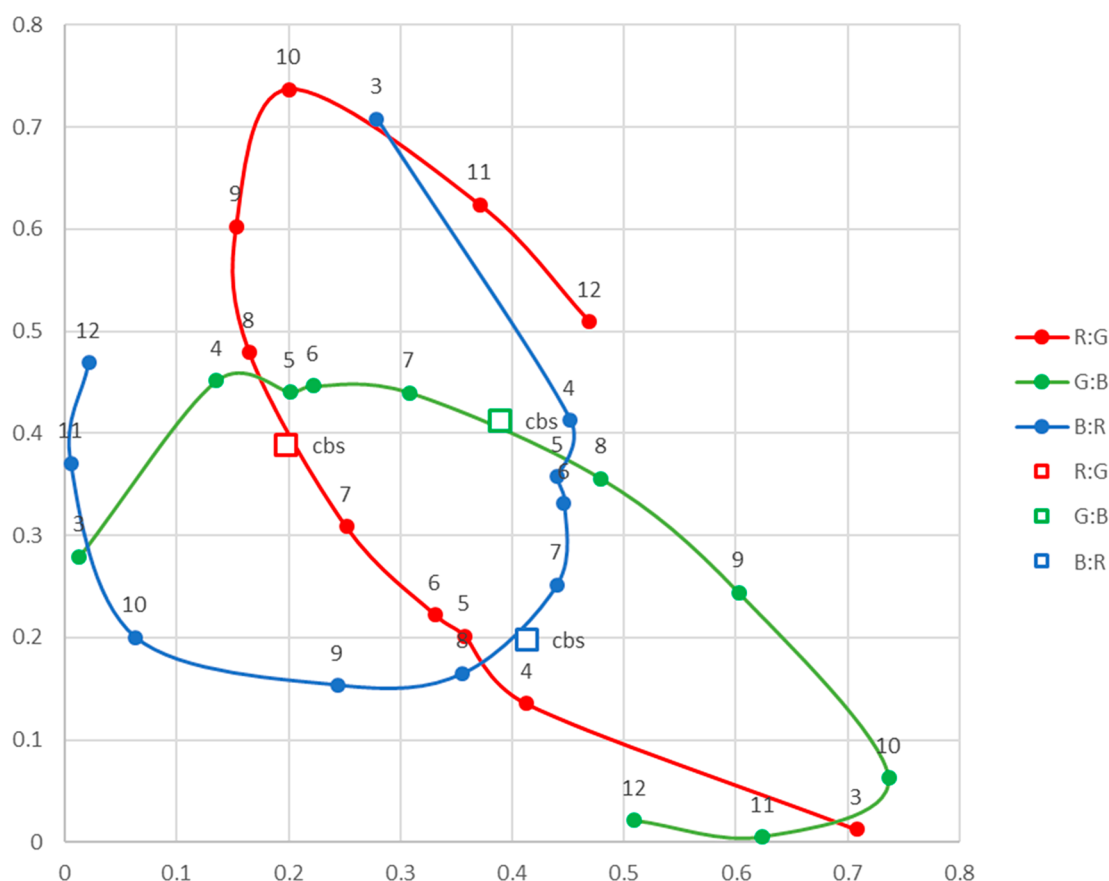


Figure 1. RGB ratios of the red cabbage pH indicator as a function of pH. R:G, G:B, and B:R ratios are shown as the red, green, and blue lines, with the pH values indicated above the close circles. The three open squares show the R:G (red), G:B (green), and B:R (blue) ratios of a sample analyzed by a student, indicating that its pH was approximately 7.4–7.6.

but not necessarily synchronous with, the presentation of concepts in lectures. We contemplated implementing changes starting with our smallest section first, and CHEM-108 is a good venue for explorations. Since USC's entire undergraduate chemistry laboratories were renovated 7 years ago in a \$12 million project when the corresponding author was the department chair, quality lab space is no longer an issue, but quality lab education still lags. There have been many innovative ideas about inquiry-based or guided-discovery laboratories,^{6,11–19} but changes required impetus. COVID-19 provided it. The laboratories described here are based on some of our on-campus experiments typical of gen chem,^{4,20–24} but the experience provided a unique opportunity for the students learning at home.

For the acid–base experiments, components a and b, the obvious choice for the acid was “white” (clear) vinegar. Store-bought vinegar is typically ~5% acetic acid by weight, which corresponds to ~0.87 M. In our on-campus laboratories, a strong base like NaOH would have been used to neutralize acetic acid and carry out titrations. At home, our choice for the base was NaHCO₃, which is sold in stores as baking soda at ~100% purity. The solubility of NaHCO₃ in water at 0 °C is 6.9 g/100 g of water,²⁵ which corresponds to ~0.82 M. Despite being only a weak base, the reaction of bicarbonate with acetic acid produces H₂CO₃, which decomposes into carbon dioxide and water.^{22–24} Therefore, the neutralization of acetic acid by a sodium bicarbonate solution can be driven to completion by warming the solution during titration to drive off CO₂ and shifting the neutralization equilibrium entirely to the right,

thereby emulating a weak acid by strong base neutralization. These components relate directly to concepts I and II. However, at home, the students faced an apparatus challenge. Before spring break, we were fully expecting to be back in session the week after, so we did not send students home with any apparatus. For these experiments, they had to improvise their own creative methods for dispensing liquids and measuring volumes. Students used measuring cups for large volumes, and medicine droppers, syringes (without needles), or homemade plastic straw “droppers” to dispense smaller volumes for their titration.

For the pH measurements, component c, red cabbage extract was the choice. The visible absorption spectrum of the anthocyanin pigment in red cabbage varies with pH over a broad range from 2 to 12,²⁶ causing the color of a solution of red cabbage dye to change from red to blue to green to yellow within this pH range. The recipe for a red cabbage pH indicator is widely known,²⁷ and the solution could be kept for up to ~3 days after preparation. The visible change in color of the indicator can be used to monitor the end point of a titration. A colorimetry analysis was employed to avoid discrepancies in individual perceptions of color, and to be able to measure pH more precisely despite not having access to a pH meter or spectrometer. In the experiments, students took photos of solutions with the red cabbage indicator at different pH values and analyzed the colors by a red–green–blue (RGB) analysis using standard image processing software (e.g., Photoshop or GIMP). Via a careful calibration of the RGB ratios against solutions with known pH values, students were

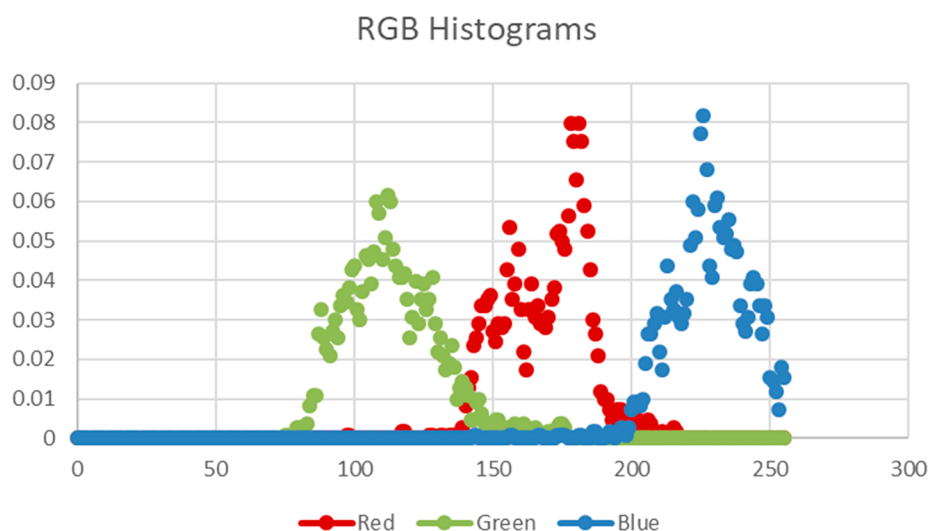


Figure 2. RGB histograms for an indicator solution at pH 6.

able to measure pH to within ~ 0.3 units, which was sufficiently accurate to assay the breakdown of a buffer for the rest of the lab. Figure 1 shows an example of this calibration. To derive this curve, store-bought vinegar with the indicator can be titrated with a standardized NaHCO_3 solution to the equivalence point, and with the concentration of vinegar now known, the pH values along the titration curve can be computed and correlated with the RGB ratios of the solution from photos taken throughout the course of the titration. For students who were unable to purchase clear white vinegar but bought apple cider vinegar or rice vinegar instead, this calibration procedure was also able to eliminate the interference arising from the intrinsic colors of their vinegar solutions, producing a fairly accurate determination of the pH analogous to white vinegar. This particular component of the experiment addresses concept I.

To illustrate the RGB analysis, Figure 2 shows an RGB histogram obtained from an indicator solution at pH ~ 6 using Gimp. Values on the horizontal axis, x , indicate the intensity level, at 256 levels from 0 to 255. Values on the vertical axis, y , represent the fraction of pixels in the image with a certain intensity level, for red, green, and blue, separately. The total intensity of red in the image, for instance, can be derived from the sum

$$I_R = \sum_{x=0}^{255} x y_R(x) / \sum_{x=0}^{255} x [y_R(x) + y_G(x) + y_B(x)] \quad (1)$$

where $y_R(x)$, $y_G(x)$, and $y_B(x)$ are the red, green, and blue histograms, respectively. The example in Figure 2 generates an R:G:B ratio of approximately 0.331:0.222:0.447, which corresponds to the following points on the calibration curves in Figure 1: (R, G) = (0.331, 0.222) on the red line, (G, B) = (0.222, 0.447) on the green line, and (B, R) = (0.447, 0.331) on the blue line, correlating to pH = 6. As the next section on the logistics of the experiments will explain, creating the procedures and the protocol for this RGB analysis was one of the team assignments for the first phase of the lab. Student design of the protocol for the RGB analysis was an integral component of the lab.

To standardize the NaHCO_3 solution without the ability to weigh out baking soda (unless students happen to have a food scale at home), students used the known solubility of NaHCO_3

in water at 0°C (6.9 g/100 g of water) to produce a saturated NaHCO_3 solution to use as a standard. Since the ice point is universal, there was no need to measure temperature at 0°C . This is an excellent point of contact with concept V concerning solid–liquid phase equilibrium and thermodynamics, where students should recognize that the equilibrium coexistence between solid and liquid water occurs uniquely at its melting temperature, $T_m = 273\text{ K}$. This part of the experiment most directly relates to concepts IV and V.

For components d and e, the students designed a buffer by determining the volume of standard NaHCO_3 solution they should use to neutralize vinegar to produce the best buffer solution. They then verified the capacity of the buffer by titrating it with NaHCO_3 to the breakdown point. The pH range of interest should be in the neighborhood of the pK_a of acetic acid, which is 4.7. For this experiment, the pH of the titrated buffer had to be measured to determine when it changed by $\sim \pm 1$ unit, and this was assessed using the RGB colorimetry analysis described above. To further assess how the capacity of the buffer changed with the quantities of acetic acid and acetate ions used, students diluted their buffers by a factor of 20 and repeated the titration to verify (a) that the pH of the buffer had not changed substantially and (b) that the buffer broke down sooner according to their predictions based on calculations using the Henderson–Hasselbalch equation. Unfortunately, there were not many options for acids to use for titrating the buffer toward more acidic pH. Lemon juice is probably the only commonly available household chemical that is more acidic than vinegar, but the color of lemon juice interferes with the RGB analysis, and we did not pursue this, though with proper calibration, this may be possible. So, in this part of the lab, which relates to concepts I, II, and III, a buffer titration was carried out using NaHCO_3 as the titrant.

For component f, the solubility experiments, acetic acid and NaHCO_3 reversed their roles. Here, vinegar was used as the titrant in order to assay the concentration of a NaHCO_3 solution, again using the red cabbage indicator to monitor the end point of the titration. The solubility of NaHCO_3 in water varies by more than a factor of 2 between 0 and 60°C , and titration by vinegar was found to be sufficiently precise to determine these variations in solubility even without a detailed RGB analysis. Some students reported they were also able to

confirm the end point by monitoring when CO_2 evolution ceased upon further addition of vinegar. Concept IV was addressed by this part of the laboratories.

Finally, for component g, determining the temperature dependence of the solubility equilibrium constant K_{sp} of NaHCO_3 required an instrument to measure temperatures between approximately 0 and 60 °C. For this, we asked our students to construct either a liquid thermometer (using store-bought isopropyl alcohol or just water) or an air thermometer at home. There are numerous recipes online for how to construct a homemade liquid thermometer, but we found that the majority of these recipes were incorrectly mistaking the thermal expansion of air for the expansion of the liquid. To properly construct a true liquid thermometer, the “bulb” must be filled entirely with liquid.²⁸ To calibrate their thermometers, students used two reference temperatures: ice point at 0 °C, which as described above could be fixed at the coexistence temperature of ice and water, as well as body temperature at 37 °C, which could be determined using a home thermometer. Using these two reference points, the temperature scale between 0 and 60 °C could be established assuming linearity. Also, to carry out the van’t Hoff analysis on K_{sp} , the solubility of NaHCO_3 was measured at a number of temperatures. Because $\ln(K_{\text{sp}})$ is plotted versus $1/T$ in the van’t Hoff analysis, neither K_{sp} nor T needed to be measured very precisely. Most students reported reasonably accurate solubility and temperature measurements and were able to extract ΔH° and ΔS° without complications. Solubility data for NaHCO_3 are known and can easily be found, so we encouraged students, if they used literature values for comparison, to cite their sources and to assess their experimental errors against the cited values. This component of the laboratories relates directly to concepts IV, V, and VI.

Throughout the experiments, the TAs worked with students persistently on the planning and execution of their experiments. While the laboratories were designed to present no safety issues, the TAs and the instructor maintained a consistent dialogue about safely with the students via Slack. Students were reminded of general safety measures, such as always performing their experiments in well-ventilated areas, exercising caution, using only water from the faucet for their hot water baths, and not exceeding 50 °C when using an isopropyl alcohol thermometer. Students were encouraged to be mindful about these factors, especially when conducting experiments on their own.

■ LOGISTICS OF THE EXPERIMENTS

Given the limited time we had during COVID-19 restrictions to prepare for the switchover to at-home laboratories, we were unable to verify all the details of our design or ascertain how well they would actually work out in the wild. However, with the concepts of the experimental design in place, we decided to leave the experiments somewhat open-ended. We designed the logistics of the experiments to divide the activities into three phases. Phase I consisted of an exploratory phase in which the students were tasked with assembling all the necessary analytical tools and developing the procedures for how to deploy these tools. Phase II consisted of the application of those tools to making buffers and assaying their capacities. The final phase, phase III, consisted of an assessment exercise where students made use of the techniques they had acquired in phases I and II in a lab test to measure ΔH° and ΔS° for the dissolution of NaHCO_3 .

Two weeks were allocated to phase I. During this phase, students were divided into three teams according to their lab sections. Each team was managed by a TA. The objective of phase I was for each team to produce a protocol, i.e. a set of instructions, for how to carry out a particular part of the bigger experiment. The first team (“team A” in the [Supporting Information](#)) was charged with developing a protocol for the neutralization of acetic acid and verifying that NaHCO_3 could be used for the stoichiometric titration of vinegar even though NaHCO_3 is a weak base. The second team (team B) was responsible for developing a protocol for the colorimetry analysis of the indicator and how to map RGB ratios to pH values. The third team (team C) was tasked with developing a protocol for how to formulate the pH indicator and validate its color variations.

During Phase I, the TAs did an intensive amount of work managing and guiding their teams to direct their students toward the goal of delivering their team protocols to be ready for the next phase of the experiments. Phase I was also critical for the entire at-home lab experience, because without any one of these protocols, none of the subsequent phases of the laboratories would be possible. Prior to the implementation of phase I, we had high confidence that the indicator formulation should present no issue. On the other hand, we were unsure whether the stoichiometric neutralization of acetic acid by NaHCO_3 was indeed possible, but we had contingency plans in case it was not. While the formulation of the colorimetric analysis was expected to be the most demanding of the three protocols, we expected any issues there to be largely technical and likely solvable. The outcomes from phase I were largely consistent with our expectations. None of the three protocols presented unsurmountable problems at the end. The biggest issue, one that we had not anticipated, turned out to be the technical accuracy of the written protocols. Writing scientifically and precisely was something freshmen students had not received a lot of training on, and the teams encountered minor issues following the instructions in each other’s protocols. Implementing a peer-review process during protocol preparation may benefit students’ technical writing skills, as well as produce clearer instructions and mitigate some of the confusion that arose during phase II. The phase I team activities were managed by the TAs using the Slack app for communications. Phase I took a few more days than the 2 weeks originally planned.

One week was allocated to the next phase, phase II, of the experiments. During phase II, students were charged with designing two buffers and validating their capacities, and the lab assignment was written to be intentionally open-ended, asking the students to design, plan, and execute their own experiments and analyze the collected data. Having responsibility over every part of their experiment, from preparation of the materials to designing the procedures, taking the measurements, and analyzing the results was something the students had limited experience with, and because of this phase II was the most challenging part of this lab experience for most of the class. Many students needed individual guidance on different aspects of phase II, particularly on the titration procedures and RGB analyses, and we provided this over Slack. We observed that being able to resolve these issues after students had struggled with them also helped students build confidence, and this prepared them for phase III of the experiments.

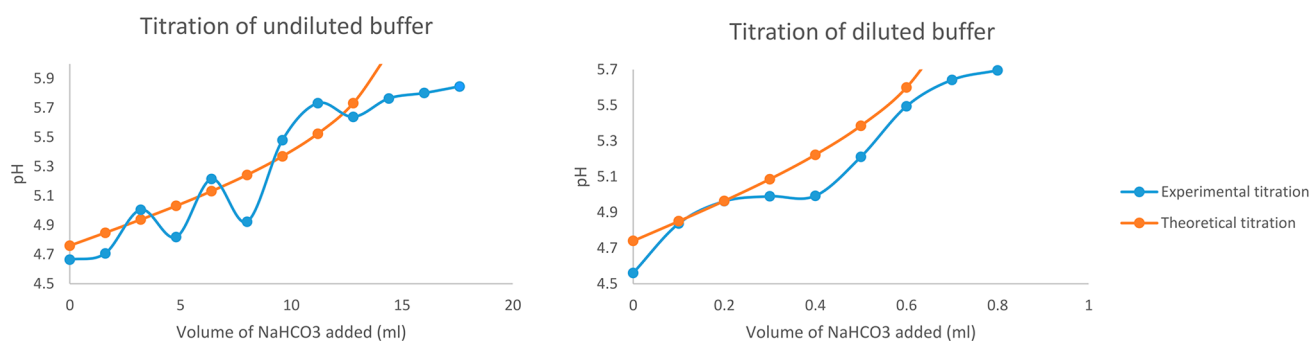


Figure 3. Student-produced experimental titration curves (blue circles) compared to computed theoretical values (orange circles) for a buffer prepared by dissolving baking soda into store-bought vinegar on the left, and the same buffer diluted by a factor of 20 on the right, where the solutions' pH measured by the RGB analysis described in the text is plotted against the volume of standardized NaHCO₃ solution added as the titrant.

In the [Supporting Information](#), we have provided a sample of both the phase I and phase II instructions. These “instructions” were written to be intentionally open-ended, to encourage students to take personal ownership of the design and construction of their own experiments. During phase I, each team understood that the success of the rest of the experiments was resting on the quality of their protocols, and that responsibility provided a positive incentive for the teams to deliver on the phase I outcomes. In particular, team B, who was in charge of the colorimetric analysis, had the most challenging assignment of all three teams, because unlike the cabbage indicator or the reaction between vinegar and baking soda, there was little to no available information on how to carry out the RGB analysis needed. Team B found ref 26, but they had to adapt a procedure that was designed for spectrophotometry to work as a colorimetric analysis, and because of this team C required more guidance and input from the instructor, who worked closely with the team over Slack and videos. The truth is the instructor himself did not know prior to phase I exactly how the colorimetric analysis would work, only that in principle it should. Phase I was therefore a discovery process for everyone in the class, students, TAs, and instructor included.

The formula that has been found to generate reliable R:G:B ratios is provided with [Figure 1](#). To generate RGB histograms in Gimp 2.10, an image of the indicator solution is imported, and an area with an approximately uniform color inside the solution is selected. The sequence of clicks, Colors > Info > Export histogram, will generate a CSV file with the RGB histograms, and applying [eq 1](#) above to each of the three color channels will produce the R:G:B ratios similar to those displayed in [Figure 1](#). [Figure 2](#) shows an example of typical RGB histograms.

To accurately measure pH, each student needed to produce his or her own calibration curves. We found that variations in camera hardware, the geometry with which they took the pictures, the particular indicator recipes they used, as well as the intrinsic colors of their store-bought vinegar solutions all had an effect on the absolute RGB values measured. Because of this, students needed to perform their own calibrations using solutions of known pH values. To achieve this, each student started with the store-bought vinegar solution and titrated it with standardized NaHCO₃ until the end point where the color of the indicator turned blue, taking pictures of the color throughout. With the original concentration of the vinegar solution now known, each student back calculated the

theoretical pH values along the titration curve, to use them to construct his/her own calibration curves. This procedure was found to produce pH measurements sufficiently consistent to be able to quantify the breakdown of a buffer in the phase II experiments. This was the most demanding component of the entire experiment, since this procedure required students to thoroughly understand every aspect of the acid/base chemistry they were learning in class, at the confluence of concepts like titrations, buffers, and the Henderson–Hasselbalch equation. This was also where students needed a lot of guidance.

During phase III, which was allocated 1 week, the students were given a lab challenge, where they were asked to utilize the experience, knowledge, and skills they had learned from phases I and II to determine ΔH° and ΔS° for the solubility equilibrium of NaHCO₃. During phase III, each student worked individually, and since this was a test, each student was responsible for every aspect of the phase III experiment. We observed that the majority of the students took a very deliberate approach to their phase III tests, spending more time designing their experiments than executing them. Overall, students reported good success with their phase III experiments.

OUTCOMES OF THE EXPERIMENT

At the end of phase III, the outcomes of the experiment were assessed using the students' lab reports, records of discussions over Slack, as well as written feedback about their overall experience submitted by the students. In the following, we describe these outcomes in three different directions: quality of the experimental results, logistic of the laboratories and their feasibility, and student learning and achievements.

While we had not expected a high degree of quantitative precision from the at-home laboratories, a good majority of the students were able to obtain qualitatively accurate results. [Figure 3](#) shows buffer titration data from one of the reports, where an undiluted buffer prepared by dissolving baking soda in store-bought vinegar was titrated with the standard NaHCO₃ solution. This is compared to the same buffer diluted by a factor of 20, whose titration is shown on the right. The target pH for both was 4.74, and the graph on the right confirms that the diluted buffer had a substantially lower capacity. While the experimental data points (blue circles) have some scatter, they qualitatively agree with the expected values (orange circles) calculated on the basis of the known concentrations of the buffer and the titrant. It is important to point out that these experiments were carried out using

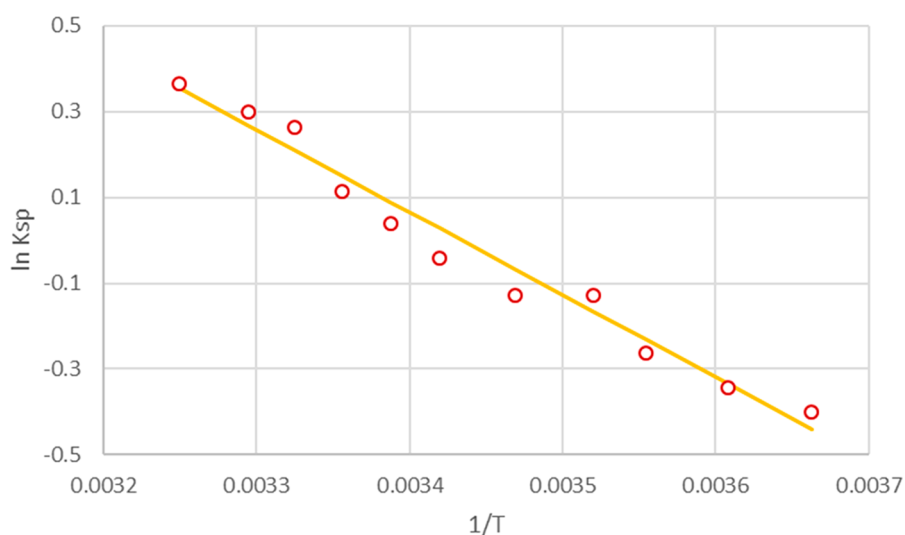


Figure 4. Van't Hoff plot of student-produced NaHCO_3 solubility data showing $\ln(K_{\text{sp}})$ versus inverse absolute temperature in units of K^{-1} .

imprecise volumetric measurements, but the results were nonetheless reasonable as long as students titrated by dispensing NaHCO_3 dropwise, which apparently produced a fairly linear relationship between drops and volume.

For the thermodynamics of the dissolution of NaHCO_3 , Figure 4 shows a van't Hoff plot of baking soda solubility data from a student report. These results were obtained by titrating a saturated solution of baking soda by vinegar, using the red cabbage indicator to visually identify the end point, where the indicator turned pink. Within this temperature range from 0 to 35 °C, K_{sp} varies by roughly a factor of 2. Over a wider temperature range from 0 to 60 °C, some students were able to observe a change in K_{sp} by a factor of ~ 4 . The magnitude of ΔH° for the dissolution of NaHCO_3 is quite small. The general observation that the solubility of baking soda changes by approximately 2-fold within this temperature range, which most students were indeed able to observe, places the ΔH° value in approximately the right range of $\sim 20 \pm 5$ kJ/mol.

Regarding the logistics and feasibility of the laboratories, phase I of the laboratories was the most labor-intensive. Each of the three TAs, who managed one of the three teams, was in constant communication with their students to make sure they worked to deliver the team protocols on time. Phase I was also the most open-ended part of the entire experiment because we were unsure whether every element of the design would work at home, since we had not carried out every part of the experiment ourselves. Of the three team protocols, the colorimetric analysis turned out to be the most challenging. That was also the part of the phase II laboratories most students had trouble with. If these laboratories were incorporated into a regular chemistry course, these issues can be mitigated by asking for a spreadsheet to be included as part of the team protocol so that the rest of the class can simply import their RGB histograms into the spreadsheet to derive the RGB ratios without having to reinvent the wheel for themselves. During all three phases of the experiment, a large volume of communications among the instructor, TAs, and students was necessary to support the students.

After phase III, written comments were collected over Slack from almost every student who was able to take part in the at-home laboratories (a small fraction of the students in the class were unable to procure all of the needed materials due to the

quarantine). Out of 44 students in the class, 35 submitted comments. A large majority of the class had an overall positive experience with the laboratories. 74% thought they had learned more from the laboratories than they would have otherwise. A similar fraction believed the laboratories improved their understanding of the concepts learned in class. 62% explicitly stated that they enjoyed this unconventional lab experience. Most preferred these laboratories to the more formulaic ones they were used to on-campus. 53% thought their laboratory skills in chemistry improved because of these at-home lab experiences. 50% reported that these experiments took more time than the on-campus laboratories, which were typically only 3 h long, with the majority of them indicating that some parts of the laboratories took them a day to several days at a time. 42% found that the results they obtained were reasonable despite the crudeness of the apparatus, and many indicated that these constraints actually helped them pay more attention to the design of their experimental procedures. About 15% felt that the technical issues they encountered detracted them from achieving the learning outcomes of the laboratories. The majority of the class felt that the at-home laboratories would be a valuable experience for a regular gen chem course. Some of the other words students used to describe their at-home lab experiences were the following: memorable, challenging, amazing, gratifying, rewarding, feel very accomplished, inspirational, most unique, fun, really cool, frustrating, confused, fascinating, and learned a lot. Some of the feedback also brought up an issue we had not anticipated. Students who were still in the dorms and others who lived in smaller spaces indicated that the experiments required quite a bit of counter surface which they did not have. For those living in the dorms, not having a sink close by was also reported to be an issue. While the majority felt that the communications among the instructor, the TAs, and the class enabled them to successfully navigate the technical issues in their experiments, the amount of communication was too much for some but not enough for others. Some students also found that balancing the time investment needed for the at-home laboratories with their other courses was challenging. Many indicated that the laboratories facilitated a positive sense of camaraderie among the class. Others felt that the at-home laboratories helped them maintain focus on their coursework and their academic lives

during the especially challenging times in the COVID-19 pandemic.

■ INTEGRATING SIMILAR EXPERIMENTS INTO A REGULAR CHEMISTRY COURSE

On the basis of the positive outcomes of this experiment, we are planning to incorporate a similar set of laboratories in the same course next year. For the next iteration, we will be assembling a lab kit, which will include a digital thermometer and volumetric apparatus, for each student to bring home. Given the experience from this year, we will be providing students with a more precise set of instructions next time but paying attention to try to not compromise the open-ended nature of the at-home laboratories too much or impede the students' latitude to explore on their own. We are also exploring the possibility of incorporating other experiments from the second-semester general chemistry course into the at-home laboratories format. In particular, some of the chemical kinetics laboratories are potential candidates.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.0c00483>.

General instructions for phases I and II (PDF, DOCX)

Phase II details (PDF, DOCX)

Phase III details (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

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